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# *Conference Report*

## *NIST WORKSHOP ON THIN DIELECTRIC FILM METROLOGY Gaithersburg, MD October 30-31, 1997*

*Report prepared by*

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## 1. Introduction

The microelectronics industry requires accurate measurements of thin dielectric films for fabrication process control, largely depending on nondestructive, in-line, optical metrology techniques such as ellipsometry and reflectometry. As device geometries continue to shrink and yield demands continue to grow, the requirement for well-calibrated, well-maintained metrology equipment becomes more critical. Demands on the equipment, demands on the metrologists, and demands on the material scientists have increased dramatically. Historically, NIST has provided the industry with the Standard Reference Materials® (SRM®) 2530 series of ellipsometrically characterized thin dielectric film standards consisting solely of $SiO_2$ thermally grown on a silicon substrate with thicknesses ranging from 10 nm through 200 nm. Because the process by which such standards are produced and characterized is costly and time-consuming, alternative pathways to ensure traceability to national standards are being sought that encompass current and near-future technology. By bringing together experts from semiconductor metrology equipment suppliers, semiconductor device producers, academia, other National Laboratories, and SEMATECH, NIST endeavored to develop a clearer understanding of both user needs and alternate approaches to reference artifacts for use in the calibration of optical process control equipment used in the fabrication of ultra thin oxides and alternate dielectric materials.

The NIST Semiconductor Electronics Division sponsored and facilitated a two-day workshop on October 30-31, 1997 at NIST, Gaithersburg, which had as its overall theme: Establishing traceability to NIST for optical metrology of thin dielectric films used in microelectronics—the evolution of reference materials for thin dielectric films. According to the 1997 National Technology Roadmap for Semiconductors (NTRS) [1], "the gate dielectric has emerged as one of the most difficult challenges for future device scaling." With this focus in mind, attendees were invited from calibration facilities of integrated circuit manufacturers, optical equipment suppliers, secondary standards suppliers, and related supporting laboratories. The format was based on presentations by invited speakers having expertise in ellipsometry, reflectometry, materials, and/or standards issues. In addition, speakers from NIST's Standards Reference Material Program (SRMP), the National Voluntary Laboratory Accreditation Program (NVLAP), the Statistical Engineering Division, and the Semiconductor Electronics Division presented several NIST approaches to traceability and standards. Finally, a summary of the NTRS and its impact on thin film metrology and standards was presented.

Two working sessions, or discussions, were held to summarize and highlight the key points from the presentations and to develop a list of action items. The first

focused on establishing a consensus definition of a standard, to determine what form it should ideally take, and what attributes of it should be certified. During the second, there was more detailed discussion of a suitable, effective, and efficient delivery method or methods for such standards that can address the needs of the industry relatively quickly. A list of action items was developed and prioritized to aid in completing the tasks. Participants volunteered to collaborate in focus groups for several of the most pressing items.

## 2. Workshop Summary

The Thin Dielectric Film Workshop was conducted over two days and divided into three technical and two working sessions. Welcoming remarks were made by David G. Seiler, Chief, Semiconductor Electronics Division, followed by several sessions in which the speakers had been invited to present specific topics within the structure of the workshop. On Thursday, October 30, Session I, Optical Metrology Techniques and Modeling and Session II, Using Standards, were held following with the working session which had a focus of developing a consensus definition of standards for film thickness measurements. Session III, Traceability from a NIST Perspective, was held, October 31. This was followed by a presentation which highlighted key challenges and goals specifically for thin dielectric film technology as reported in the 1997 NTRS. The workshop ended with an extended working session during which several action items were discussed and agreed upon.

### 2.1 Session I: Optical Metrology Techniques and Modeling

The workshop began with presentations which offered overviews of the two most commonly utilized optical metrology techniques used during the fabrication of semiconductors, single-wavelength ellipsometry (SWE) and reflectometry. These were given by Harland G. Tompkins of Motorola, Inc. and Richard Brown of Nanometrics, Inc. Tompkins reminded the participants that ellipsometry does not directly measure film thickness but requires the use of a layer model for interpretation, and that while single-wavelength ellipsometry is capable of very high precision, it is primarily a one-film, one-substrate technique with an uncertainty that increases as film thickness decreases. He also highlighted the dichotomy between (1) the fabrication-facility user of ellipsometry who focuses on measurement consistency but is uninterested in models being used; and (2) the in-house calibration lab "guru," or the instrument manufacturer, both of whom must understand how to alter the model for new or modified film growth processes.

While reflectometry is generally utilized in a thicker film regime than gate dielectrics, the reflectometer continues to be a common tool used in process control measurements. Brown elaborated on the fact that it has been widely used in thin-film metrology and noted current efforts to include spectroscopic ellipsometry as an integral part of the reflectometer. He indicated that customers are becoming much more interested in accuracy in addition to the traditional emphasis on repeatability. Brown also described correlative measurements between reflectometry, single-wavelength ellipsometry, and spectroscopic ellipsometry to support the development of secondary standards at Nanometrics, Inc. With the ever-decreasing feature size and gate dielectric thickness, and the implementation of complex dielectric films, fabrication facilities are turning to spectroscopic ellipsometry for a more detailed examination of the optical properties. John Woollam, J. A. Woollam Co., Nhan Nguyen, Semiconductor Electronics Division, NIST, and David Aspnes, North Carolina State University, discussed instrumentation, measurement, and modeling considerations when using spectroscopic ellipsometry.

Woollam emphasized that three factors must be addressed simultaneously for accurate thin-film thickness measurements: samples, instrumentation and data analysis. He elaborated on such factors as understanding the complexity of the films being measured, their interface structure and roughness, the need for proper and consistent cleaning procedures, the need for accuracy in all instrument angle settings and in wavelength calibration, the stability of source and detectors, and finally, the proper selection of material structure model and goodness-of-fit criteria. He also illustrated his points with a number of response surface plots showing the interplay between the ellipsometric parameters, $\Delta$ and $\psi$, choice of angle of incidence and errors in its setting, and sensitivity to changes in film thickness in the analysis process. He concluded with the observation that the choice of the optical model for the material has a significant effect on results and is a major reason for discrepancies between laboratories. Some of the most difficult tasks involve unambiguously determining the optical properties of the substrate materials used in manufacturing. This particular point was reiterated several times in later talks and in the ensuing discussions. Optical properties play an important role in the modeling calculations which determine the thickness and refractive index of the dielectric films.

Nguyen illustrated this point when he discussed a variety of models on a single set of measurement data for a range of thin oxide films, attesting to the marked

differences in results based on the choice of substrate and oxide dielectric functions made at the outset of the analysis. All these choices fit the data with the same goodness-of-fit criteria, but resulted in a range of index and oxide thickness values outside the range acceptable for current technology needs.

Aspnes dealt in some detail with the issue of obtaining reference quality dielectric functions of the silicon substrate, discussing the physics of surface termination of the crystal bonds and how it modifies the ability to obtain the true bulk dielectric function. He reviewed various experimental and theoretical approaches to obtaining true bulk values and noted a technique for establishing a hydrogen-terminated surface for <111> silicon which appears to give the best results to date; unfortunately, there is no known comparable procedure for the technologically more important <100> surface of silicon. He also noted that there is a state of confusion among research results concerning the transition region between silicon and $SiO_2$ films, but his best optical evidence is that it is approximately 0.7 nm thick.

## 2.2 Session II: Using Standards

According to the 1997 NTRS, it is "critically important to have suitable reference materials available when a measurement is first applied to a technology generation, especially during early materials and process tool development" [1]. John Panner, IBM, Burlington Vermont, spoke to the use and application of Standard Reference Materials in an industrial fabrication facility. Panner observed that often the requirements of industry exceed current capabilities of national reference standards and explained how effective "work-around" solutions such as the development of in-house standards for thinner and thicker films are implemented. He described the process of using these standards for the critical operation of "tool matching" (e.g., ensuring that measurements on a given metrology tool give results that are consistent with those from the variety of metrology tools used in various stages of processing) which is required because a process must have a consistent analysis regardless of the tool being used. He noted the need for standards in a wide range of materials and thicknesses, characterized over a wide range of wavelength, and pointed out the importance of being able to qualify analysis algorithms since the algorithms must be thought of as an inherent part of the measurement tool. Richard Spanier, Rudolph Technologies, Inc., gave a perspective of how standards were used by the instrument manufacturers in the set-up and calibration of the tools in the fabrication facility and the importance in using them to determine that the tool continues to work correctly in order to monitor important processes. He further asserted that it is critical for the manufacturer to be able to distinguish whether it is the process or the process monitoring equipment which has malfunctioned, causing an apparent loss of control of the process. To resolve this uncertainty, reference artifacts play a critical role, and for current technology needs, they must have long term stability of $\pm 0.015$ nm.

At the close of Session II, a working session was conducted so that participants could try to reach a consensus on the definition and form of an appropriate thin film standard. It was noted that the desire for traceability to NIST is being driven by International Standards Organization (ISO) requirements and NTRS goals. Use of elaborate optical models to obtain the most accurate values of film thicknesses turned out not to be an overwhelming concern at present. However, one of the major concerns and one of the most critical issues regarding standards for thin-film metrology is the stability of thin films. Neither SRMs nor secondary standards for $SiO_2$ are demonstrably stable at the nearly 0.01 nm level needed to support optical tool performance verification, or tool matching required by current semiconductor manufacturing technology. A request was made that NIST identify films that might be stable at this level, or develop cleaning procedures that could retrievably bring films such as $SiO_2$ back to this level. Suggestions for a wider range of thicknesses, both thicker and thinner than standards currently offered, non-$SiO_2$ and possibly film-free silicon, were also made. Another important topic of discussion was a need for educating the end-user of both metrology equipment and standards. It was suggested that NIST and SEMATECH could make significant contributions in this area by assuming the leadership role.

## 2.3 Session III: Traceability from a NIST Perspective

NIST personnel presented an overview of the thin dielectric film Standard Reference Material effort, the Standard Reference Material Program, the NVLAP, and the expertise provided by the Statistical Engineering Division during this session which began on the second day of the workshop.

NIST responded to a need for optically characterized thin dielectric film standards when it began its program to develop (1) the high-accuracy ellipsometer, (2) artifacts which became the Standard Reference Material 2530 series, and (3) an elaborate analytical protocol which would offer the industry the most accurate representation of a "true" thickness of $SiO_2$ on a silicon substrate. The standard, as issued, is limited in effectiveness because in reaching for that accuracy, the model and specific method of ellipsometric measurement are

not readily transferrable to commercial systems and situations. Barbara Belzer presented the NIST SRM 2530 series development, highlighting NIST's desire to provide accurate measurements and the recognition to change its perception in order to stay in step with the industry it serves. She discussed the alternatives to applying a NIST Traceable Reference Material (NTRM) and other collaborative methods emphasizing "thinking outside the box" to develop relevant and effective standards.

Nancy Trahey explained the overall philosophy of the Standard Reference Material Program, which is to "quantify the accuracy and compatibility (traceability) of measurements and measurement processes." She explained that NIST is federally mandated in its responsibility for national measurement infrastructure, and as such has developed rigorous processes for developing and certifying NIST SRMs, a time-consuming and labor-intensive activity. Trahey also illustrated NIST's ability to address standards concerns that require a more timely response by describing the concept of an NTRM as it applies to the gas cylinder industry.

The NVLAP provides accreditation of calibration or testing laboratories to perform calibrations or tests within a specified scope of accreditation. Douglas Faison of NVLAP outlined the program and described the rigorous evaluation, based on ISO Guide 25, that a laboratory must undergo in order to become accredited. He also described several alternatives when national standards are not available to support the traceability requirements of the accreditation process.

Keith Eberhardt of the Statistical Engineering Division at NIST presented the analysis of a two-laboratory experiment comparing measurements from NIST's High-Accuracy Ellipsometer with those from VLSI Standards, Inc. on a series of $SiO_2$ films ranging from nominally 5 nm to 1000 nm. The results of this experimental collaboration were both the generation of specific data to support traceability for secondary $SiO_2$ standards beyond the range of NIST SRMs and experience gained regarding use of two-laboratory testing to establish traceability to NIST. Eberhardt illustrated the complexity of initially establishing the uncertainty level between two measurement systems for a set of artifacts given an underlying uncertainty about sample stability. Based on his analysis, he offered suggestions for improvements in the experimental design. These included, but were not limited to, the incorporation of statistical control procedures (control charts) and the additional exchanges to increase the number of degrees of freedom for use in the statistical analysis.

Jim Greed, VLSI Standards, Inc., highlighted the technical challenges described in the 1997 NTRS. Perhaps the most important issue is the "challenge of providing measurement techniques and standards for which the uncertainties in measurement are smaller than the process tolerances." In addition, the importance of appropriate and readily available reference materials for application when a measurement is initially employed in a fabrication facility cannot be overemphasized. Greed spoke to the possible need for new materials and "new metrics" in order to keep pace with the needs of the microelectronics industry.

### 2.4 Summary Discussion and Action Items

The final discussion revolved around determining a suitable and effective delivery mechanism for national standards for thin gate dielectric materials. This resulted in a list of several action items described below in order of their relative priority.

Based on experience illustrated in the analysis of the NIST-VLSI Standards experiment, the majority of the participants believed that an exchange using both real and simulated ellipsometric data would be useful in detecting the differences that may occur in calculations based on the algorithms applied during analysis in conjunction with inexact material models or minor errors in setup conditions. NIST will organize the data and distribute them to collaborating participants, then summarize and distribute the results. Further action is dependent upon the results of this study, which will take place early in calendar year 1998.

There was an overall affirmation that a thin dielectric film NTRM program could serve as a viable means for maintaining thin-film standards and keeping them current with industry needs. Regardless of the delivery mechanism, the critical requirement that certified standards must be stable, the ability to maintain this stability or return the film thickness to its original value was a prominent topic during discussion. Development and publication by NIST of an effective cleaning procedure was encouraged. It was suggested that a round-robin in conjunction with specific cleaning procedures be conducted after NIST completes an initial study to test procedures for their "transportability." It is also highly desirable to determine what the common contaminants on the "standards" might be using a variety of methods such as Auger, mass spectrometry, and some chemical analysis tests following cleaning. It was felt that contaminants may vary depending on storage conditions and a environmental factors at individual locations.

It was also agreed that it is critical that NIST develop routine Spectroscopic Ellipsometry Measurements characterizing thin-film standards to support the rapidly growing numbers of spectroscopic instruments in use in fabrication facilities.

Barbara Belzer of NIST, Gil Yetter of SEMATECH, Jon Opsal of Therma-Wave, Clive Hayzeldon of KLA/Tencor, Jim Chiles of SGS Thompson, and Jay Jellison of Oak Ridge National Laboratories agreed to serve as the steering committee for a Thin Dielectric Film Working Group, created during the workshop, which will collaborate to stay focused on issues pertinent in industry.

Educating the user of thin-film standards in common metrology methods and the care and cleaning required to maintain traceable standards was a concern shared by all participants. NIST and SEMATECH were asked to investigate possible training arrangements.

Because maintaining a stable and retrievable zero thickness artifact is extremely difficult, a suggestion was made to conduct a so-called process round robin for hydrogen-terminated <111> Si. In this experiment, everyone would create their own sample according to a common recipe and use spectroscopic ellipsometry for characterization.

## 3. References

[1] The National Technology Roadmap for Semiconductors, Semiconductor Industry Association, San Jose, CA (1997).